

# OD-77160 /80

6a

77160 C/44 E17 A41 G02 FARB 03.04.79  
BAYER AG \*DT 2913-218  
03.04.79-DT-913218 (23.10.80) C07c-67/08 C07c-69/54 C07d-319/06

Prepn. of (meth)acrylic acid ester(s) - by esterification of (meth)acrylic acid with polyhydric alcohol(s) in presence of phosphite ester and a phenol

In the prodn. of (meth)acrylic acid esters by esterification of (meth)acrylic acid (I) with an alcohol(s) (II) (from 2- to 4-hydric satd. aliphatic alcohols and their oxyethylation prods. or 1-3 C alkyl mono- or disubst. 5-hydroxymethyl-dioxan-1,3), the reaction is carried out under azeotropic conditions in the presence of (a) a (cyclo)aliphatic and/or aromatic hydrocarbon with a b.pt. of 40-120°C; (b) an esterification catalyst, (c) polymsn. inhibitor, (d) 0.001-5 wt.% (w.r.t. I+II) of an organic ester of phosphorous acid (III) and (e) 0.01-0.3 wt.% (w.r.t. I + II) of a mono- or dihydric phenol (IV).

#### ADVANTAGE/USE

High yields of the esters are obtd. without polymer formation of discolouration and without serious redn. in reactivity of the resulting monomer. The esters have good storage stability, low acid value and little inherent smell and are useful raw materials for electron beam curing lacquers, UV curing printing inks, coating compsns.

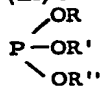
A(1-D10) E(5-G8, 5-G9A, 7-A4, 10-E2D, 10-E2E, 10-G2A) G(2-A2C, 2-A4A) N(4-C).

15

moulding and casting compsns., etc.

#### DETAILS

(III) is e.g.



where R, R' and R'' are  
1-8 C alkyl, 2-4 C hydroxy-alkyl, 2-4 C haloalkyl, aryl (esp. phenyl) or 1-4 C alkyl-subst. aryl. Pref. 10-60%

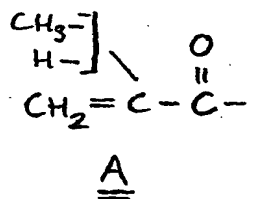
of the (III) is added before azeotropic esterification, and the remainder is added continuously during the esterifn., e.g. with a carrier gas such as air or N.

#### EXAMPLE

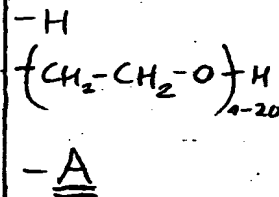
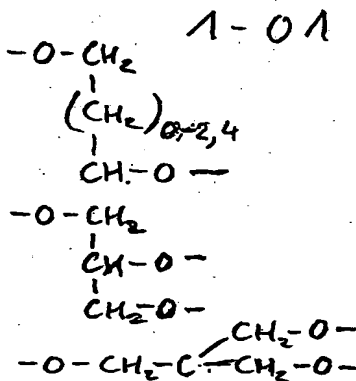
A mixt. of 3.5 kg acrylic acid, 5.36 kg oxyethylated trimethylolpropane (OH value 550 mg KOH/g), 2.7 kg cyclohexane, 0.073 kg conc. H<sub>2</sub>SO<sub>4</sub>, 0.006 kg triethylphosphite (V) and 0.004 kg 2,5-di-tert.butylhydroquinone (VI) was subjected to azeotropic esterification for 17 hrs. at 82°C with sepn. of water whilst 10 l/hour air satd. with (V) was led through. The mixt. was cooled and 0.0024 kg (VI), 0.005 kg (V) and 0.002 kg toluhydroquinone added, and the

DT2913218+

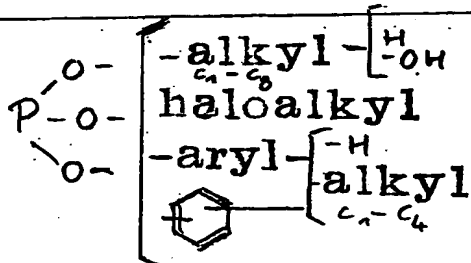
cyclohexane distilled off under vacuum. 50 l/h air satd. with (V) was then passed through at a sump temp. of 105°C and a press. of 50 mbar. The prod. was filtered to give a monomer prod. with an acid value of 2.5, an iodine colour value of 0-1 and a viscosity of 120 mPas.(19pp513)



15



DT2913218



Referate aus CENTRAL PATENTS INDEX von DERWENT

VERSCHLOSSELT

KORRIGIERT

BEMERKUNGEN

BLATT-NR.

BLATTZAHL

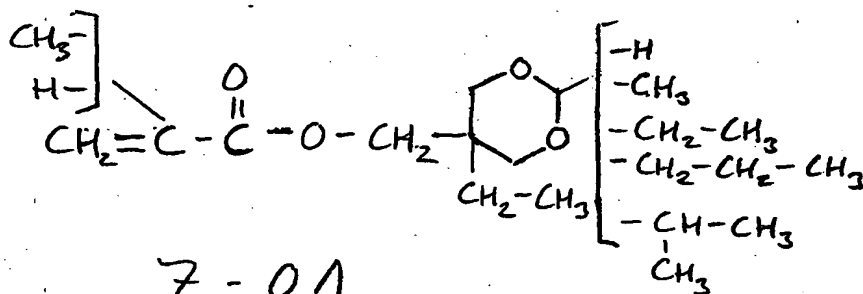
J.v. Lamprecht WARTH

1

2

BEST AVAILABLE COPY

22.



7-01

$$\text{CH}_2-\text{OH}$$

Diethyleneglycol 8-02

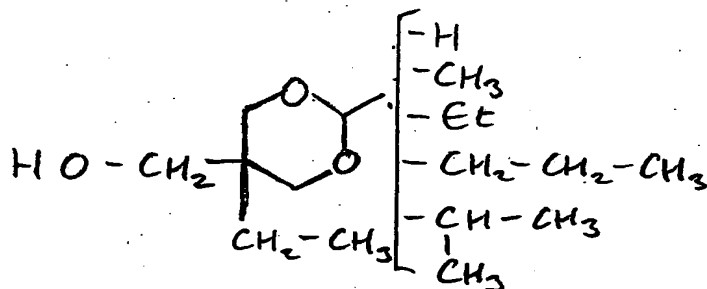
$$\text{CH}_2-\text{OH}$$

Glycerin 9-02

2-02 Pentaerythrit 10-02

2-02

Pentacrythrit 10-02



17-02